



# **Nuclear Rocket Ceramic Metal Fuel Fabrication Using Tungsten Powder Coating and Spark Plasma Sintering**

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## **LIST OF ACRONYMS AND SYMBOLS**

ANL	Argonne National Laboratory
CERMET	ceramic metal
CIF	Center Innovation Fund
CVD	chemical vapor deposition
dUO <sub>2</sub>	depleted uranium dioxide
EDS	energy dispersive x-ray spectroscopy
GE	General Electric
HIP	hot isostatic pressing
LeRC	Lewis Research Center
MSFC	Marshall Space Flight Center
NCPS	Nuclear Cryogenic Propulsion Stage
NERVA	Nuclear Engine for Rocket Vehicle Application
NTP	nuclear thermal propulsion
NTR	nuclear thermal rocket
SEM	scanning electron microscopy
SPS	spark plasma sintering
TD	theoretical density
TP	Technical Publication
UO <sub>2</sub>	uranium dioxide
W	tungsten
WPC	tungsten powder coating



## NOMENCLATURE

$M$	mass
$M_A$	mass of component A
$M_B$	mass of component B
$V$	volume
$V_A$	volume of component A
$V_B$	volume of component B
$V_T$	total volume
$v_A$	volume fraction of component A
$v_B$	volume fraction of component B
$\rho$	density
$\rho_A$	density of component A
$\rho_B$	density of component B
$\rho_{\text{mix}}$	density of the mixture
$\rho_W$	density of tungsten



## TECHNICAL PUBLICATION

# NUCLEAR ROCKET CERAMIC METAL FUEL FABRICATION USING TUNGSTEN POWDER COATING AND SPARK PLASMA SINTERING

### 1. INTRODUCTION

NASA's goal to conduct exploration missions beyond low-Earth orbit, including manned Mars missions, forces the nation to develop new enabling technologies. Nuclear thermal propulsion (NTP) is a technology innovation that has the potential to enable manned Mars missions. Nuclear thermal rockets (NTRs) utilize nuclear thermal energy to generate propulsive forces and offer a higher specific impulse than traditional chemically propelled spacecraft (liquid engines, solid motors, etc.), which reduces transient times and limits astronaut exposure to space radiation. A schematic of an NTR can be seen in figure 1.

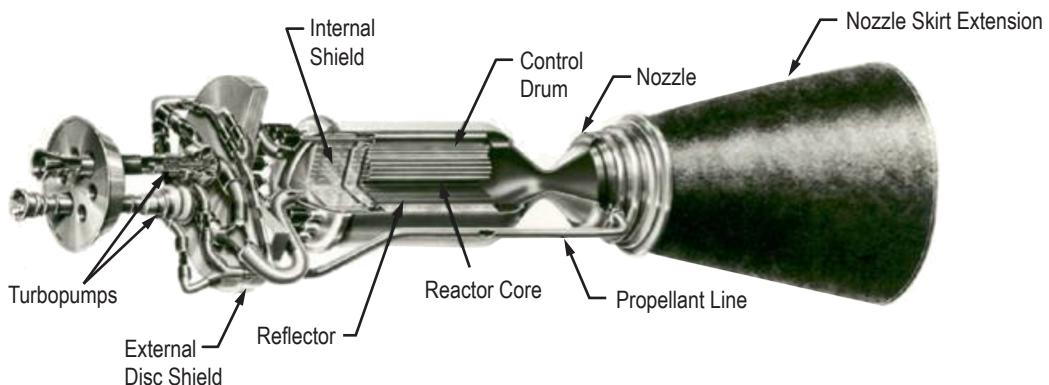


Figure 1. NTR schematic.

Efforts to develop NTRs have been ongoing intermittently since the 1960s; however, no manned-rated NTRs currently exist. Key to the development of an NTR is a robust nuclear fuel material that can perform in a harsh, high-temperature ( $>2,200$  K) hydrogen environment. In the 1950s, 1960s, and 1970s, two types of nuclear rocket fuel materials emerged from extensive research efforts—graphite-based fuels and ceramic metal (CERMET) fuels. The Rover/Nuclear Engine for Rocket Vehicle Application (NERVA) program successfully demonstrated the feasibility of NTRs by conducting a series of reactor development and nuclear engine system tests.<sup>1,2</sup> Rover/NERVA NTRs used nuclear fuel composed of a graphite matrix impregnated with uranium carbide; however, the graphite-based fuels employed in the Rover/NERVA engines suffered from fuel erosion. An alternative fuel type that also received attention in the 1960s and 1970s was a refractory

metal-based fuel consisting of a tungsten (W) matrix with imbedded uranium dioxide ( $UO_2$ ) ceramic fuel particles. This fuel type is typically designated as a CERMET fuel. CERMET, for the purposes of this Technical Publication (TP), refers to W- $UO_2$  fuels. The challenges that material scientists and engineers have faced while attempting to develop W- $UO_2$  CERMET fuels have been documented and redocumented. This TP outlines a new technique to mitigate fabrication challenges using tungsten powder coating (WPC) and spark plasma sintering (SPS). WPC is a new process, developed internally at NASA Marshall Space Flight Center (MSFC) for coating  $UO_2$  particles. SPS is an innovative powder consolidation technique. This research was conducted under a fiscal year 2016 MSFC Center Innovation Fund (CIF) project with additional support from the NASA NTP project.

## 2. BACKGROUND

The research conducted during this CIF project leverages both past research efforts from the 1960s and 1970s and more recent research conducted at MSFC around 2012–2015. This section summarizes the important aspects of past research and identifies its link to this CIF effort.

### 2.1 Heritage Programs

Over the last 50 years, scientists and engineers have explored many methods of CERMET fuel fabrication. Research into the fabrication of CERMET fuel (tungsten and depleted uranium dioxide ( $d\text{UO}_2$ )) was undertaken by General Electric (GE), Argonne National Laboratory (ANL), and NASA Lewis Research Center (LeRC) in the 1960s and 1970s.<sup>3–5</sup> Under the GE710 program, GE developed a process to consistently fabricate CERMET fuel elements by first cold pressing and then hydrogen sintering blended  $\text{W-UO}_2$  powders to form fuel wafers. Then, the wafers were stacked and subjected to hot isostatic pressing (HIP) to form elements. The GE710 process was instrumental in demonstrating the viability of CERMET fuels. Fuel elements fabricated during the program survived tens of thousands of hours of nonnuclear and in-pile testing.<sup>3,4</sup> ANL CERMET fuels research focused on fabricating full-length fuel elements (45.7 cm) using a HIP process. Fuel elements created in this fashion commonly failed to meet dimension tolerances. LeRC explored multiple fabrication approaches including various forms of hot pressing, an extrusion technique, roll compaction, and cold pressing/sintering. Some of these methods showed potential; however, no fabrication technique reached full maturity under the program. While these heritage programs did not result in the release of a CERMET fuel specification or outline a proven process for fabrication, each program contributed to understanding the challenges (i.e., failure mechanisms, feedstock requirements, etc.) faced in CERMET fuel fabrication, paving the way for future research. A discussion of these challenges is presented in section 2.3 of this TP.

### 2.2 Recent Research at Marshall Space Flight Center

Under the Nuclear Cryogenic Propulsion Stage (NCPS) project (2012–2015), MSFC engineers and scientists labored to produce a 16-in CERMET fuel element. Research included successful HIP fabrication of surrogate CERMET fuel elements and attempts to develop a coating method for fuel particles. The need for coated fuel particles was established by past research efforts.<sup>6</sup> Efforts under the NCPS project led to the development of an experimental chemical vapor deposition (CVD) system to deposit a protective tungsten coating on  $\text{UO}_2$  fuel particles prior to fuel element fabrication.<sup>7,8</sup> The complexity of the CVD coating process forced MSFC researchers to explore alternative coating methods, which led to the development of WPC. WPC is a relatively simple process that uses a binder to adhere tungsten powder to the surface of  $\text{UO}_2$  particles.

MSFC's research focused mainly upon a full-length HIP process as the sintering/consolidation method because of the possibility of producing entire fuel elements in one sintering step. HIP

cans shown in figures 2 and 3 were fabricated and filled with a blend of W-d<sub>2</sub>O<sub>2</sub>. Filled cans were subjected to a HIP process to consolidate the powder within.

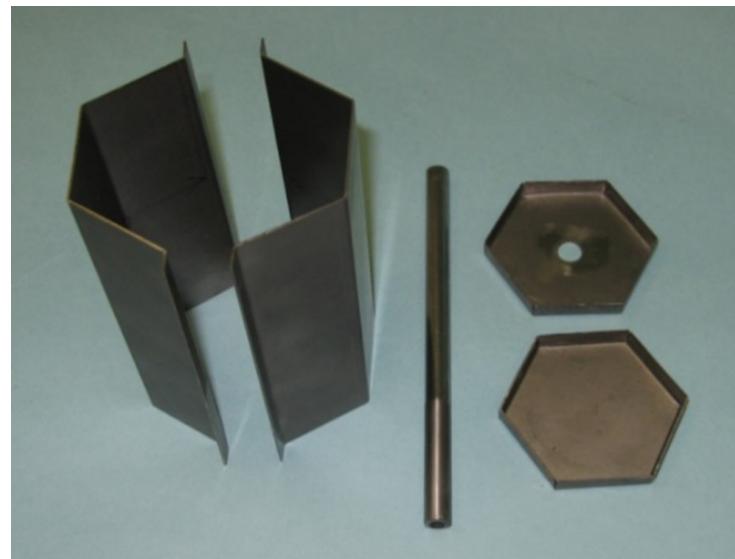


Figure 2. HIP can components.



Figure 3. Filled HIP can.

Figure 4 shows a surrogate (does not contain dUO<sub>2</sub>) element post-HIP. Tungsten-dUO<sub>2</sub> elements fabricated using this full-length HIP process suffer from low packing densities (less than 85% theoretical density (TD)) which lead to distortion of the fuel elements and fracturing of HIP cans.<sup>9</sup> Researchers realized the potential of beginning the fuel element fabrication process with a fully dense fuel wafer. Exploring a process to fabricate dense (greater than 85% of TD) wafers created an interest in SPS, a process that uses electric current and pressure to rapidly sinter powder into dense consolidated material. Limited research has been conducted to evaluate SPS fabrication of CERMET fuels. SPS fabrication using WPC UO<sub>2</sub> fuel particles has never been researched.

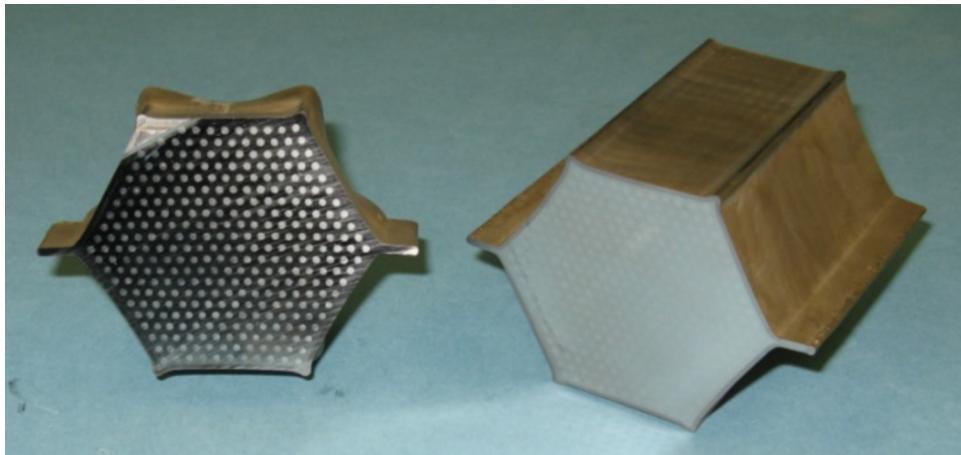


Figure 4. Surrogate elements post-HIP.

### 2.3 Ceramic Metal Fabrication Challenges

Challenges faced by MSFC researchers stem from the starting material or feedstock quality. A stable fuel material requires a high-quality feedstock. Requirements for quality feedstocks were specified by heritage programs.<sup>6</sup> Namely, UO<sub>2</sub> particles that are spherical, 10–100  $\mu$  in diameter, and contain low levels of impurities (less than 300 ppm) are required to produce fuel elements with suitable material properties. This CIF project used the highest quality feedstocks available; however, the material did not meet the specifications identified by heritage programs. (See section 4.1 for material specifications used for this research.)

The pursuit of appropriate feedstock material is ongoing. In addition to starting with an adequate feedstock material, heritage programs identified the need to coat fuel particles. The coating serves as a protective cladding that prohibits the hydrogen propellant from easily reacting with the UO<sub>2</sub> particles. The coating also aids in the formation of a uniform microstructure which is key to the stability of the fuel. Fuel elements fabricated from low-quality feedstocks that are uncoated or poorly coated suffer from agglomeration and segregation which causes the fuel material to fail prematurely.

Figure 5 depicts agglomeration of particles based upon coating method and demonstrates how the microstructure improves with coating quality. For uncoated particles, the micrograph shows agglomeration, plenty of fuel particle-to-particle contact, and poor fuel particle distribution within the tungsten matrix. CVD-coated particles are uniformly distributed and do not suffer from agglomeration, while powder-coated particles exhibited less agglomeration and a more uniform distribution than uncoated particles. Figure 6 is a photo of a HIP can cross section depicting material that suffers from segregation. The majority of the dUO<sub>2</sub> (darker colored material in this image) has migrated to the edges near the wall of the HIP can and the tungsten (lighter colored material) remained in the center. This material will not have the appropriate mechanical strength, which is provided by the tungsten matrix in W-UO<sub>2</sub> CERMETs. The dUO<sub>2</sub> around the edges will rapidly dissociate into free uranium and oxygen when exposed to hydrogen at elevated temperatures, resulting in rapid fuel loss. The research conducted under this CIF project sought to fabricate high-density fuel material (greater than 90% of theoretical) and to improve fuel particle distribution within the tungsten matrix. Fuel material with these characteristics will mitigate UO<sub>2</sub> dissociation/instability in hydrogen due to the negative effects of agglomeration and segregation and will improve mechanical strength.

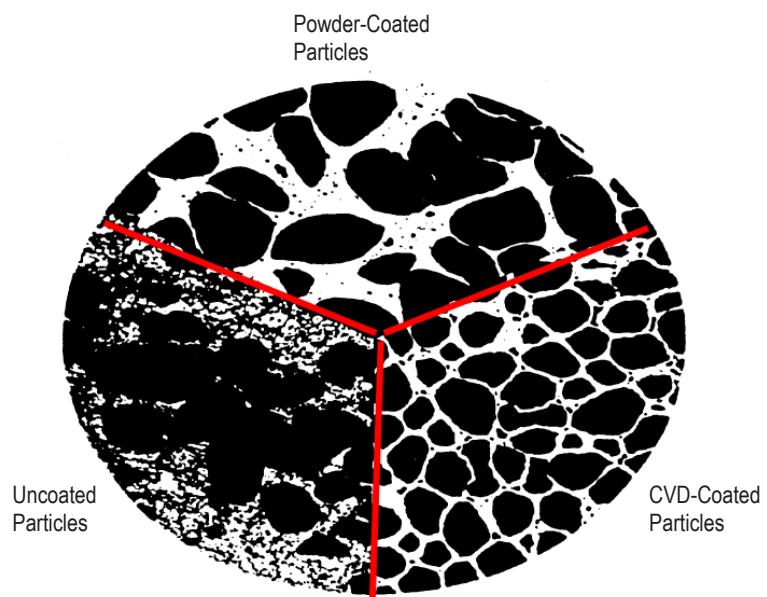


Figure 5. Particle coating effects.

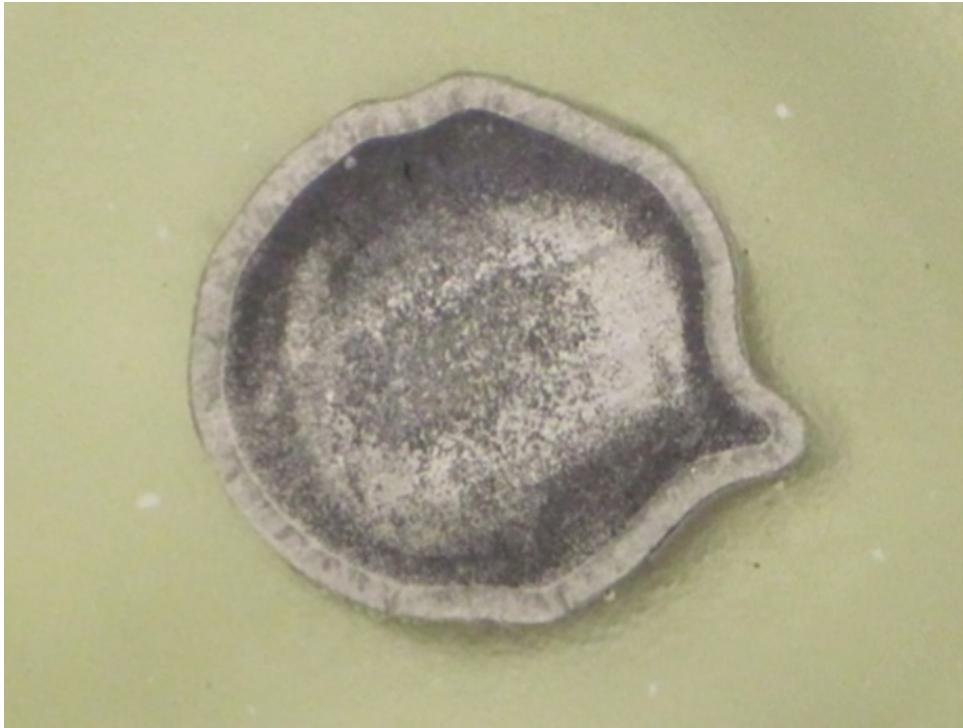


Figure 6. Segregation in HIP can.

#### 2.4 Mitigation Approach

This research uses WPC and SPS to mitigate the challenges that were identified by previous research. WPC uses an organic binder that is mixed with the blended powders. During the mixing process, the binder pastes the smaller tungsten particles to the surface of the larger fuel particles coating the dUO<sub>2</sub> particles with tungsten. Figures 7 and 8 are micrographs of WPC particles. Agglomeration and particle-to-particle contact is less likely to occur when particles are completely encapsulated with tungsten. SPS was used to address distortion and low density issues that occurred during the full-length HIP process explored under the NCPS project. As mentioned in section 2.2, SPS is a sintering technique that uses pulsed electrical current to consolidate powders into solid material. Powder material is loaded into a graphite die. Current is passed through the die and the material, while a mechanical ram applies an axial load to the material. The current causes Joule heating which elevates the temperature to the material's sintering temperature. Sintering occurs when a material at an elevated temperature in powder form becomes soft and coalesces at a molecular level. The post-sintered material is a solid mass instead of a blend of loose powders. SPS is a rapid process that sinters powdered materials in minutes instead of the hours or days that are required for conventional hydrogen/furnace sintering techniques that were employed during the GE710 program.

### 3. OBJECTIVES

This project sought to evaluate the feasibility of WPC and SPS to produce a robust nuclear fuel material. The primary goal is to further the development of CERMET fuels by demonstrating a viable method to produce fuel material, which is fundamental to the production of a prototypic fuel element for NTR applications. WPC provides the protective tungsten coating, and SPS is used to consolidate the blended powder ( $\text{W-dUO}_2$ ) material. The fabrication process for this research builds on the GE710 process by beginning with dense wafers or compacts that could be stacked to form fuel elements. WPC simplifies the fuel particle coating process, and SPS allows for the rapid sintering of blended fuel material into highly dense wafers. Future research, which is beyond the scope of this TP, will seek to develop a process for fabricating fuel elements of specific geometries and detail from dense wafers.

The density and microstructure of the sintered wafers were evaluated to provide data to predict the material performance capability. Microstructure (i.e., fuel particle agglomeration, segregation, and distribution within the tungsten matrix) and density are important characteristics needed to assess material performance. Stable fuel material requires fuel particles that are uniformly dispersed within the tungsten metal matrix displaying little or no contact between fuel particles. Furthermore, fuel material shall be dense without excess pores or voids within the tungsten matrix, which reduces the mechanical strength of the fuel material. Experiments were developed to evaluate these requirements for fuel material fabricated via SPS using WPC  $\text{dUO}_2$  feedstock material.

## 4. EXPERIMENTAL PROCEDURES

### 4.1 Material

For this research, tungsten was purchased from H.C. Starck. Particles sizes ranged from 9 to 13  $\mu$  with a 0.1 ppm maximum iron, oxygen, nitrogen, and carbon. The depleted  $\text{UO}_2$  particles were supplied by Oak Ridge National Laboratory. Particles were greater than 150  $\mu$ . Depleted uranium dioxide and tungsten were blended to yield a W-60 vol%  $\text{dUO}_2$  mixture. The heritage mixture ratio 60 vol%  $\text{dUO}_2$  is routinely employed when conducting CERMET research.

### 4.2 Tungsten Powder Coating Process

Quantities of W- $\text{dUO}_2$  were weighed and blended in 100-g batches. This batch size was driven by the WPC process. Each 100-g batch contained approximately 54 g of tungsten and 46 g of  $\text{dUO}_2$ . A small quantity of an organic binder material was added to each batch. Then, each batch was blended in a Turbula® shaker-mixer for approximately 1 hour. The blended material was further processed to ensure that the smaller tungsten particles were bounded to the surface of the larger  $\text{dUO}_2$  particles. A micrograph of tungsten powder-coated material can be seen in figure 7. The two figures show smaller tungsten particles affixed to the surfaces of much larger particles.

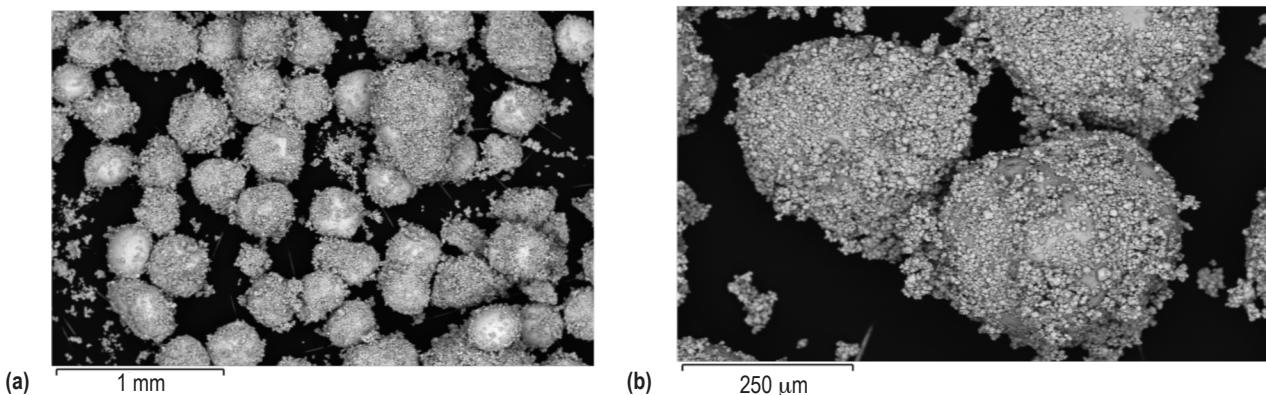


Figure 7. WPC micrograph: (a)  $\times 1$  and (b)  $\times 200$ .

### 4.3 Spark Plasma Sintering Process

Approximately 31 g of blended W-60 vol%  $\text{dUO}_2$  material were loaded into a 20-mm graphite die and sandwiched between two graphite punches. Thirty-one grams were chosen to produce post-sinter specimens that were approximately 6 mm thick, which made convenient rough density calculations possible prior to making more precise density measurements. This graphite die/punch assembly

was loaded into the SPS system. Sintering was accomplished by increasing the temperature at a rate of 100 °C per minute and pressure at a rate of 20 MPa per minute until the material reached the pre-determined sintering temperature and pressure (sintering pressure = 50 MPa for all specimens). The sintering temperature and pressure were maintained for 20 minute; then, the material was cooled at a rate of 20 °C per minute. The sintering temperature was varied as shown in table 1. Each specimen was given a unique identification number as shown in figure 8.

Table 1. SPS process parameters.

No. of Specimens	Sintering Temperature (°C)	Sintering Pressure (MPa)
1	1,600	50
4	1,700	50
4	1,750	50
5	1,800	50
1	1,850	50

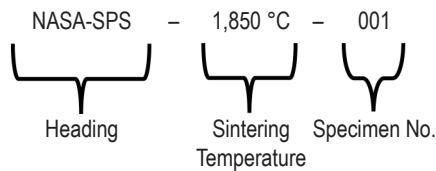


Figure 8. Specimen identification scheme.

A total of 15 cylindrical specimens approximately 20 mm in diameter and 6 mm thick were fabricated. Figure 9 shows macroscopic images of NASA-SPS-1800C-001. Specimens appear to be fully dense from a macroscopic prospective. No voids or cracks were visible on the surface nor was there any evidence of segregation. Cross sections did not reveal any macroscopic subsurface defects.

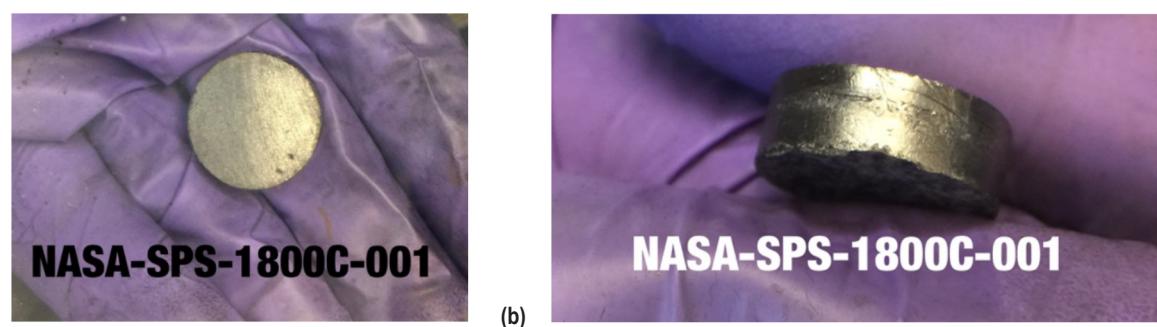


Figure 9. Specimen NASA-SPS-1800C-001: (a) top view and (b) side view.

## 5. RESULTS

### 5.1 Density

The density of each specimen was measured twice using Archimedes' technique. An average density for each specimen is listed in table 2 along with a comparison between the average measured density and the TD, which was  $14.282 \text{ g/cm}^3$  (see sec. 5.1.1). All specimens exhibited density measurements above 95% of TD. A plot of density versus temperature is provided in figure 10, showing an identifiable trend of density increase with increasing temperature.

Table 2. Specimen density data.

Specimen	Thickness (mm)	Diameter (mm)	Average Density (g/cm <sup>3</sup> )	TD (%)
NASA-SPS-1850C-001	5.90	19.93	14.20	99.46
NASA-SPS-1800C-001	5.45	19.95	14.06	98.47
NASA-SPS-1800C-002	5.94	19.96	14.07	98.57
NASA-SPS-1800C-003	5.57	19.91	14.06	98.48
NASA-SPS-1800C-004	6.03	19.91	14.03	98.26
NASA-SPS-1800C-005	5.60	19.93	14.03	98.24
NASA-SPS-1750C-001	6.10	19.89	14.09	98.68
NASA-SPS-1750C-002	6.15	19.90	14.01	98.15
NASA-SPS-1750C-003	5.60	19.96	14.09	98.70
NASA-SPS-1750C-004	5.70	19.90	14.10	98.73
NASA-SPS-1700C-001	6.00	19.90	14.00	98.06
NASA-SPS-1700C-002	6.40	19.93	14.01	98.11
NASA-SPS-1700C-003	5.93	19.90	13.94	97.63
NASA-SPS-1700C-004	6.00	19.96	14.02	98.19
NASA-SPS-1600C-001	6.10	19.90	13.87	97.18

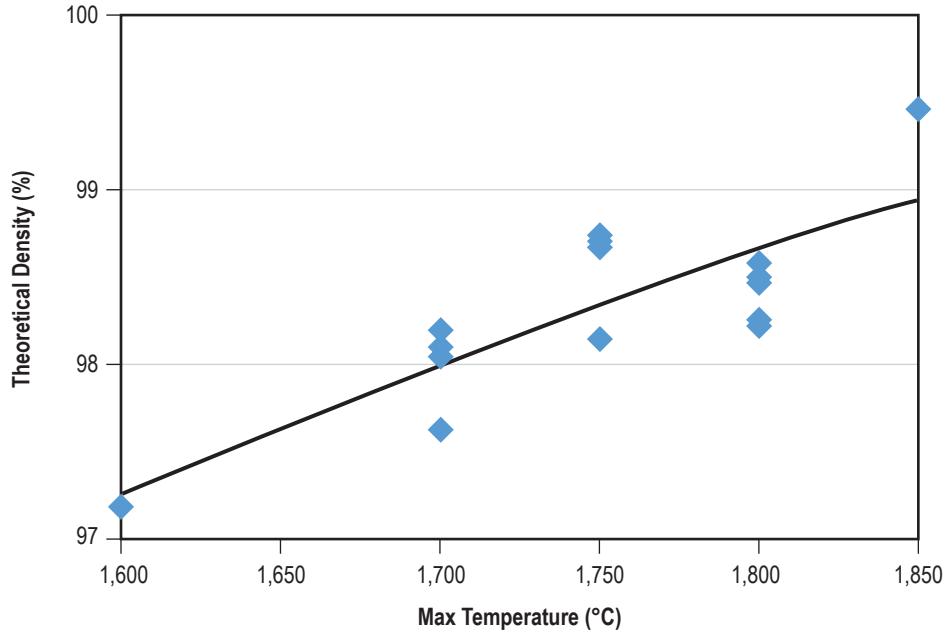


Figure 10. Plot of TD versus temperature.

### 5.1.1 Theoretical Density

As previously stated, TD is the maximum achievable density value that would be obtained if the specimen's particles were perfectly arranged and consolidated with no porosity. Though TD is most optimal, it is rarely obtained; therefore, the specimen's measured density values are typically compared to the TD as an indication of sintering efficiency. For example, a specimen with a TD of 20 g/cc and a measured density of 19.5 g/cc has a density that is approximately 98% theoretical. The rule of mixtures was used to determine the TD of the W-60 vol% dUO<sub>2</sub> mixture:

$$\rho = M / V . \quad (1)$$

The density of a mixture is as follows:

$$\rho_{\text{mix}} = (M_A + M_B) / V_T , \quad (2)$$

where

- $\rho_{\text{mix}}$  = density of the mixture
- $M_A$  = mass of component A
- $M_B$  = mass of component B
- $V_T$  = total volume.

The rule of mixtures is derived as follows:

Rearranging equation (1) yields:

$$M = V * \rho . \quad (3)$$

Substituting equation (3) into equation (2) yields equation (4):

$$\rho_{\text{mix}} = (V_A * \rho_A) + (V_B * \rho_B) / V_T . \quad (4)$$

Using the relationship that the volume of component A ( $V_A$ ) divided by  $V_T$  is the volume fraction of component A ( $v_A$ ) and the volume of component B ( $V_B$ ) divided by  $V_T$  is the volume fraction of component B ( $v_B$ ) yields equation (5), the rule of mixtures:

$$\rho_{\text{mix}} = v_A * \rho_A + v_B * \rho_B . \quad (5)$$

Figure 11 shows how the rule of mixtures was used to calculate the TD for the W-dUO<sub>2</sub> specimens. The TD is equal to 14.282 g/cm<sup>3</sup>. The tungsten density is  $\rho_W$ .<sup>10</sup> The density of dUO<sub>2</sub> is  $\rho_{\text{dUO}_2}$ .<sup>11</sup> Vol% dUO<sub>2</sub> is the volume percentage of dUO<sub>2</sub> in the mixture, and the  $\rho_{\text{TD}}$  is W-60 vol% of the dUO<sub>2</sub> mixture.

$$\rho_W = 19.25 \text{ gm/cm}^3 \quad \rho_{\text{dUO}_2} = 10.97 \text{ gm/cm}^3 \quad \text{vol\%dUO}_2 = 60\%$$

$$\rho_{\text{TD}} = (1 - \text{vol\%dUO}_2) * \rho_W + \text{vol\%dUO}_2 * \rho_{\text{dUO}_2} = 14.282 \text{ gm/cm}^3$$

Figure 11. TD calculation.

## 5.2 Microstructure

Scanning electron microscopy (SEM) was used to characterize the microstructure of multiple specimens. Micrographs were obtained from a JEOL JSM-6610LV SEM. Specimens were mounted in epoxy. Some specimens were sectioned prior to mounting to expose the subsurface microstructure. Specimen preparation for SEM included grinding and polishing to produce a smooth contamination-free surface. Both secondary electron and backscatter images were obtained. Elemental analysis was performed via energy dispersive x-ray spectroscopy (EDS) using an EDAX silicon drift detector.

The dispersion of d $\text{UO}_2$  within the tungsten matrix can be seen in figures 12 and 13 for NASA-SPS-1800C-005 and NASA-SPS-1750C-004. The majority of the fuel particles are surrounded by tungsten providing minimal particle interconnectivity. The tungsten metal matrix is completely sintered with minimal pores or voids. The fuel particles are spherical and nearly monodisperse.

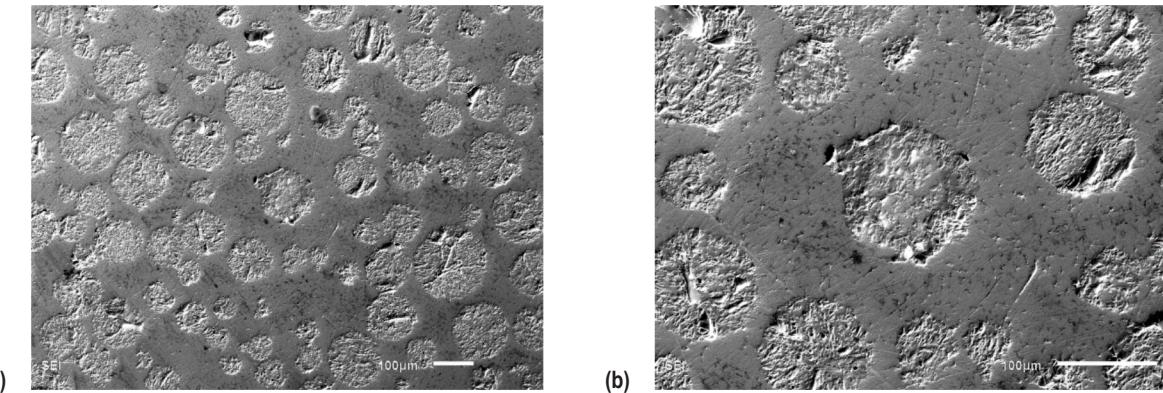


Figure 12. NASA-SPS-1800C-005 micrograph: (a)  $\times 100$  and (b)  $\times 250$ .

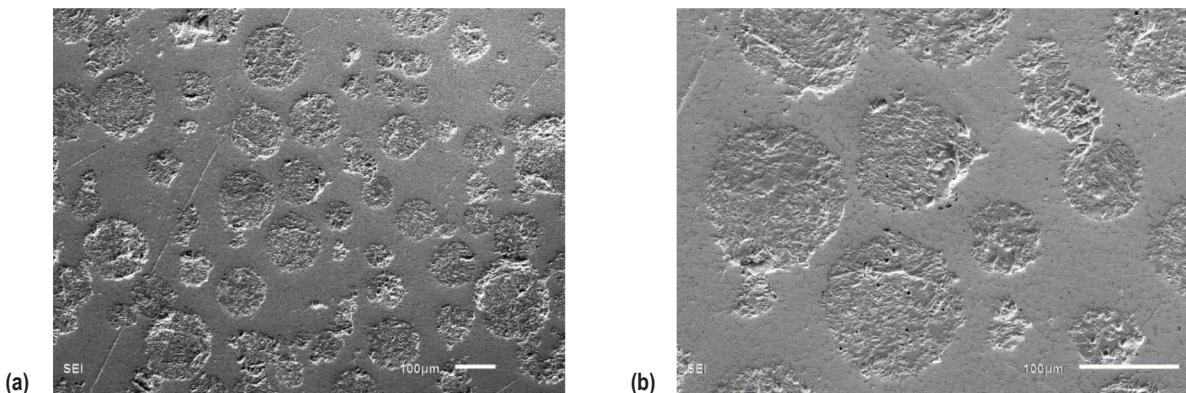


Figure 13. NASA-SPS-1750C-004 micrograph: (a)  $\times 100$  and (b)  $\times 250$ .

A cross section of NASA-SPS-1800C-005 can be seen in figure 14. The cross section reveals some particle elongation which is likely due to axial loading during the SPS process. Evidence of particle pullout was also observed in some micrographs. Pullout can be accredited to specimen preparation (grinding).

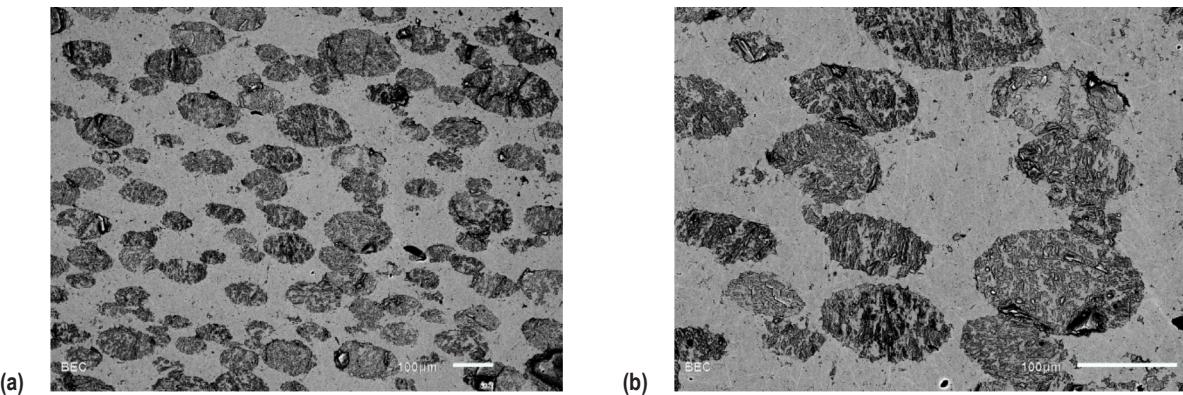


Figure 14. NASA-SPS-1800C-005 cross-section micrograph: (a)  $\times 100$  and (b)  $\times 250$ .

EDS analysis (fig. 15) provides a very distinctive image of the material's microstructure. Smaller flakes of imbedded d<sub>UO</sub><sub>2</sub> can be seen within the tungsten matrix. The flakes were likely formed during the initial Turbula® blending process.

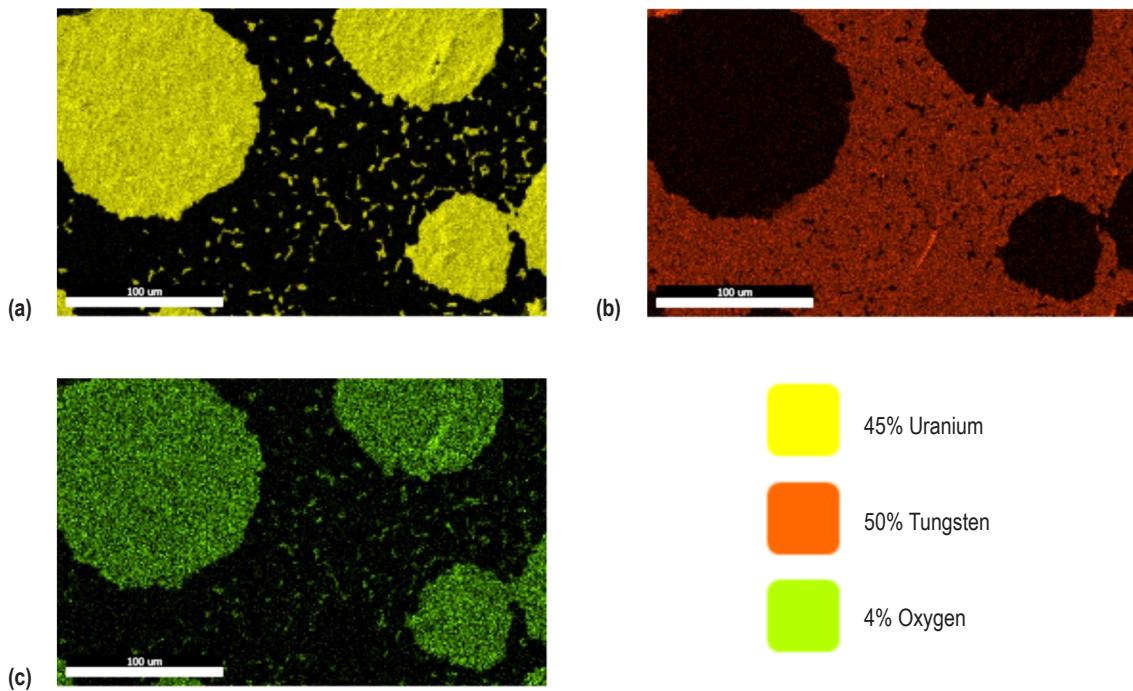


Figure 15. NASA-SPS-1800C-005: (a) uranium, (b) tungsten, and (c) oxygen.

## 6. CONCLUSIONS

All technical objectives were achieved. SPS and WPC can potentially be employed to produce W-d $\text{UO}_2$  CERMET fuel wafers that are dense and that have a desirable microstructure. Density values for fuel wafers that were fabricated using the method described herein measured above 95% of the TD. Density values in this range coincide with wafers with little to no voids as was revealed by the included microscopy and elemental analysis. The microstructure was uniformly dispersed with tungsten metal surrounding most of the fuel particles.

More research is needed to fully assess this material. A study of the interface between d $\text{UO}_2$  particles and the tungsten matrix is forthcoming and will be included in future research. Future research will also include hot hydrogen testing to assess material properties and performance. Furthermore, a measure of the material hardness would provide more insight into the mechanical properties of the material. Research to study the microstructural effects of fuel particle size and fuel loading would also be insightful.

The ultimate goal is to develop a repeatable process for producing a robust CERMET fuel element that can be used for NTR applications, however, much research is needed to achieve that goal. Fuel elements for current NTR design concepts range in length from 40 to 70 cm or more. An NTR could require in excess of 100 elements per reactor core. While SPS provides a method to rapidly produce fuel wafers of varying geometries (heritage NTR fuel elements are typically hexagonally shaped), an SPS system capable of producing longer fuel wafers would likely be required. A process could be developed using GE's 710 approach (GE710) of stacking SPS wafers and then hot isostatic press-bonding the wafers to form the full length element. Possibly, a custom SPS system capable of producing a 20-cm-long wafer could be produced. Custom dies for producing hexagonal SPS fuel wafers have already been the subject of other research projects.<sup>9</sup> Future research is needed to address these and other issues that are beyond the scope of this TP.

## APPENDIX A—RAW DATA

Tables 3 and 4 show the raw data from each specimen in this TP.

Table 3. Raw data table 1.

Specimen Identifier	Fabrication Date	Thickness (mm)	Diameter (mm)	Initial Mass (g)	Mass With GRAFOIL® (g)	Mass After GRAFOIL Removed (g)
NASA-SPS-1800C-001	5/11/2016	5.45	19.95	31.40	28.17	24.17
NASA-SPS-1800C-002	5/11/2016	5.94	19.96	31.40	28.39	26.02
NASA-SPS-1800C-003	5/11/2016	5.57	19.91	31.41	28.54	24.42
NASA-SPS-1800C-004	5/11/2016	6.03	19.91	31.41	28.44	25.98
NASA-SPS-1800C-005	5/11/2016	5.60	19.93	31.41	28.41	24.45
NASA-SPS-1750C-001	6/9/2016	6.10	19.89	31.43	28.38	26.10
NASA-SPS-1750C-002	6/13/2016	6.15	19.90	31.40	28.50	26.31
NASA-SPS-1750C-003	6/16/2016	5.60	19.96	31.41	28.48	24.48
NASA-SPS-1750C-004	6/20/2016	5.70	19.90	31.40	28.51	24.18
NASA-SPS-1700C-001	6/13/2016	6.00	19.90	31.40	28.42	25.69
NASA-SPS-1700C-002	6/22/2016	6.40	19.93	31.40	28.53	27.68
NASA-SPS-1700C-003	6/30/2016	5.93	19.90	31.40	28.44	25.34
NASA-SPS-1700C-004	7/6/2016	6.00	19.96	31.40	28.52	25.61
NASA-SPS-1600C-001	6/30/2016	6.10	19.90	31.40	28.43	26.30
NASA-SPS-1850C-001	7/11/2016	5.90	19.93	31.40	28.31	25.03

Table 4. Raw data table 2.

Specimen Name	Water Temp (°C)	In Air No. 1 (g)	In Water No. 1 (g)	Density No. 1 (g/cm <sup>3</sup> )	In Air No. 2 (g)	Density No. 2 (g/cm <sup>3</sup> )	In Water No. 2 (g)	Average Density (g/cm <sup>3</sup> )	Percent of TD (g/cm <sup>3</sup> )
NASA-SPS-1800C-001	23.8	24.23	22.51	14.07	24.22	22.50	14.05	14.06	98.47
NASA-SPS-1800C-002	23.6	26.05	24.21	14.08	26.06	24.21	14.06	14.07	98.57
NASA-SPS-1800C-003	23.6	24.46	22.73	14.08	24.47	22.73	14.05	14.06	98.48
NASA-SPS-1800C-004	23.6	26.02	24.17	14.04	26.02	24.17	14.02	14.03	98.26
NASA-SPS-1800C-005	23.6	24.48	22.74	14.03	24.49	22.75	14.02	14.03	98.24
NASA-SPS-1750C-001	22.6	26.09	24.25	14.10	26.10	24.25	14.08	14.09	98.68
NASA-SPS-1750C-002	22.6	26.30	24.43	14.03	26.31	24.44	14.00	14.01	98.15
NASA-SPS-1750C-003	23.2	24.48	22.74	14.09	24.48	22.74	14.09	14.09	98.70
NASA-SPS-1750C-004	23.2	24.18	22.47	14.10	24.18	22.47	14.09	14.10	98.73
NASA-SPS-1700C-001	22.6	25.69	23.86	14.02	25.70	23.86	13.98	14.00	98.06
NASA-SPS-1700C-002	22.6	27.68	25.71	14.03	27.69	25.71	13.98	14.01	98.11
NASA-SPS-1700C-003	23.0	25.34	23.53	13.94	23.35	23.53	13.93	13.94	97.63
NASA-SPS-1700C-004	23.2	25.61	23.79	14.02	25.61	23.79	14.02	14.02	98.19
NASA-SPS-1600C-001	23.2	26.30	24.16	13.89	26.04	24.16	13.86	13.87	97.18
NASA-SPS-1850C-001	23.2	25.03	23.27	14.21	25.03	23.28	14.19	14.20	99.46

## APPENDIX B—IMAGES

### B.1 Photographs

Figure 16 shows photographs of the SPS samples.

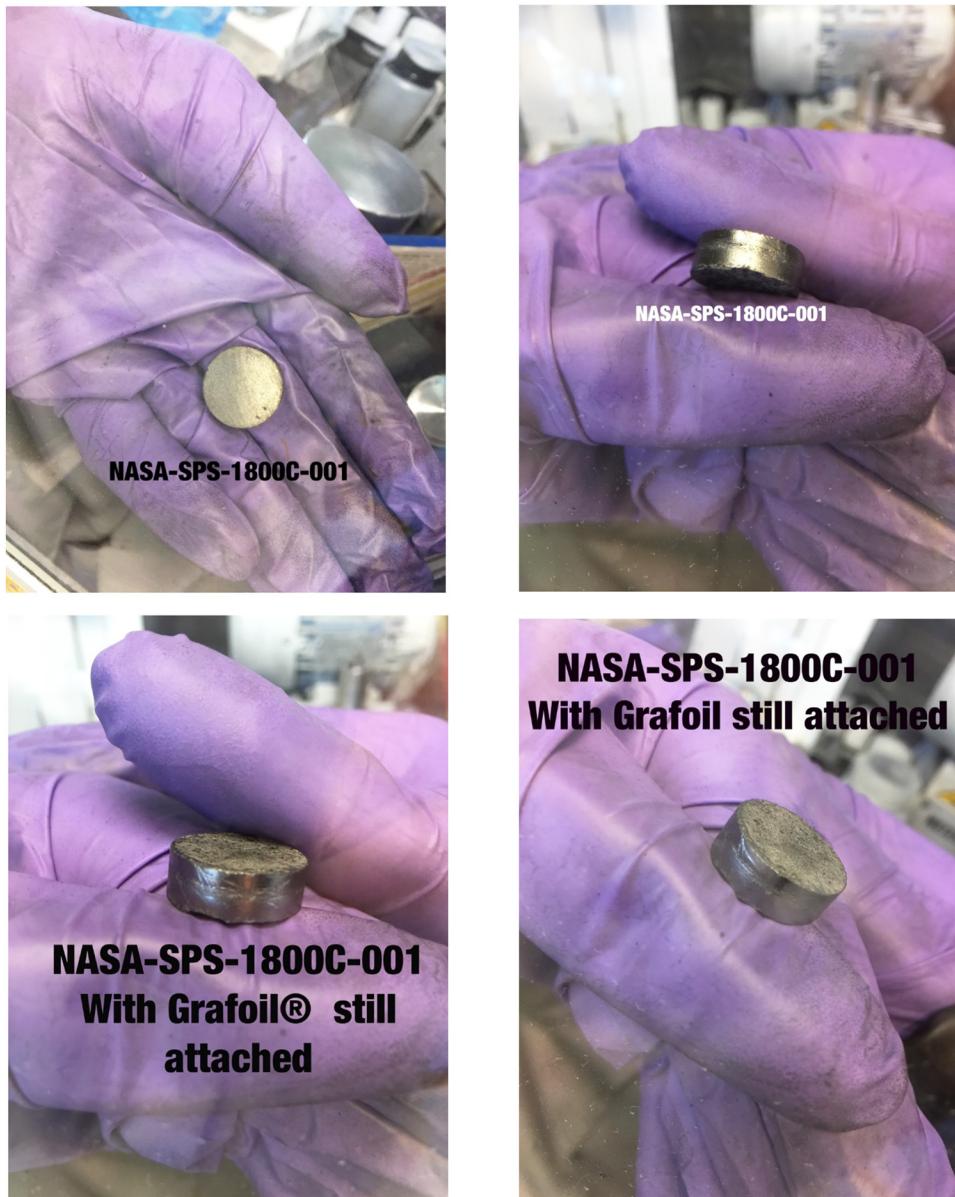


Figure 16. Photographs of NASA-SPS-1800C-001 and NASA-SPS-1800C-002 samples.

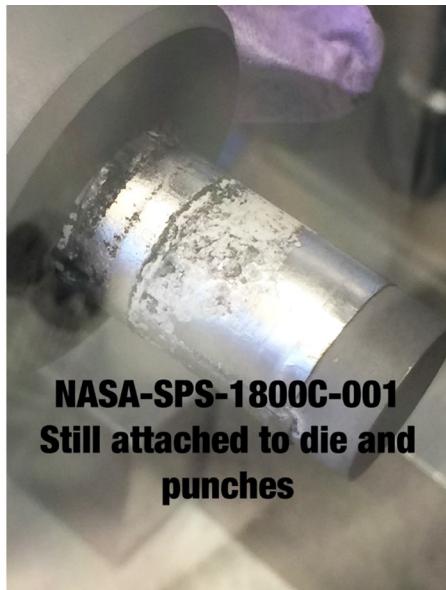


Figure 16. Photographs of NASA-SPS-1800C-001 and NASA-SPS-1800C-002 samples (Continued).

## B.2 Scanning Electron Microscopy Images

Figure 17 shows SEM images of the SPS samples.

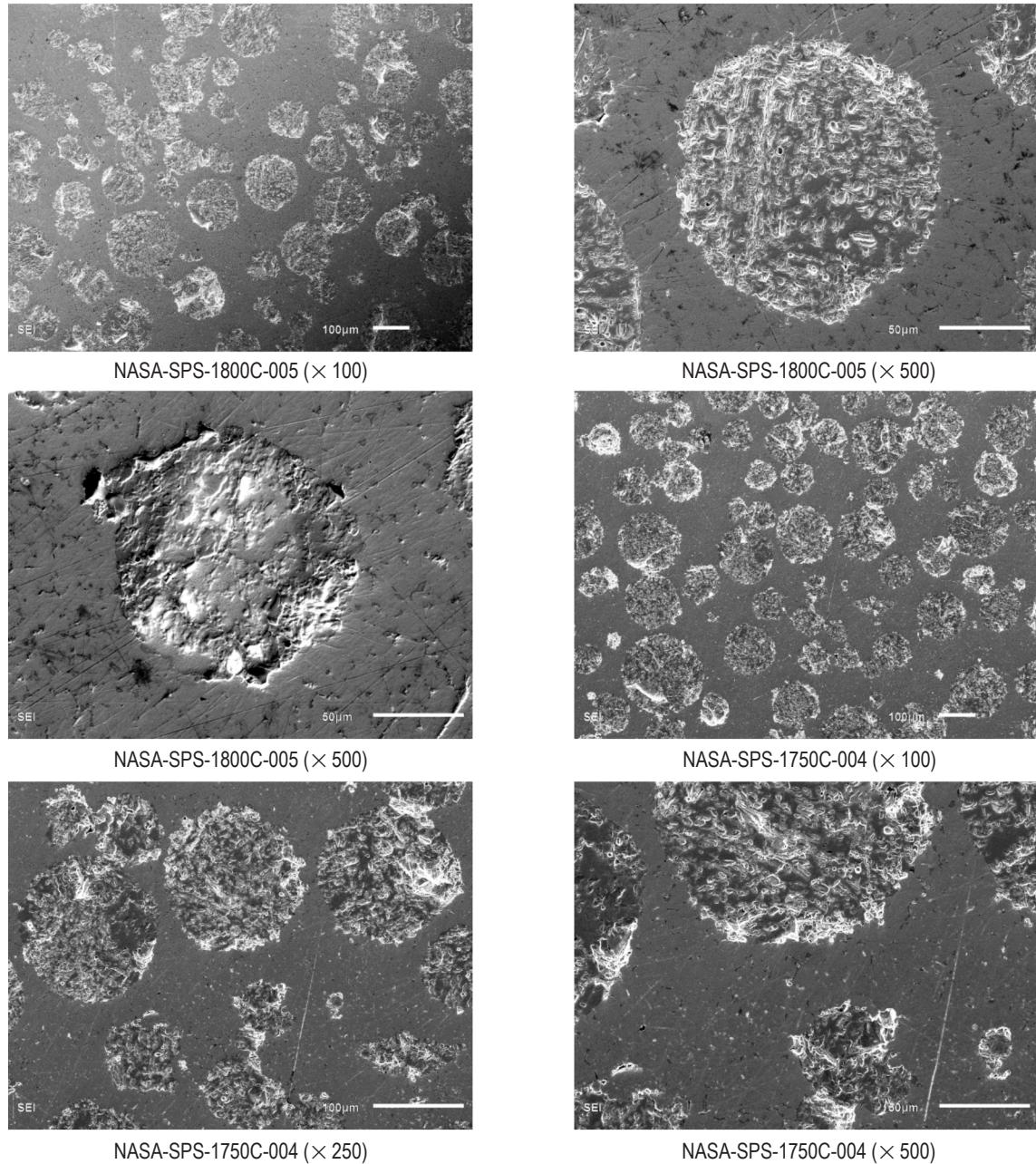
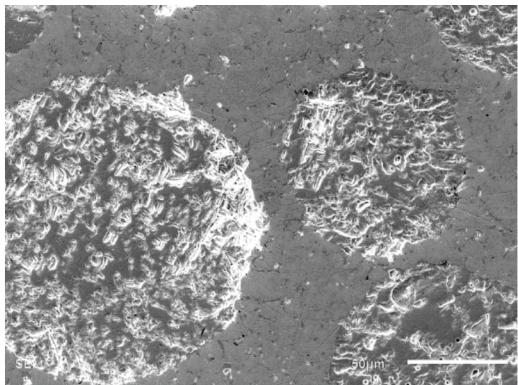
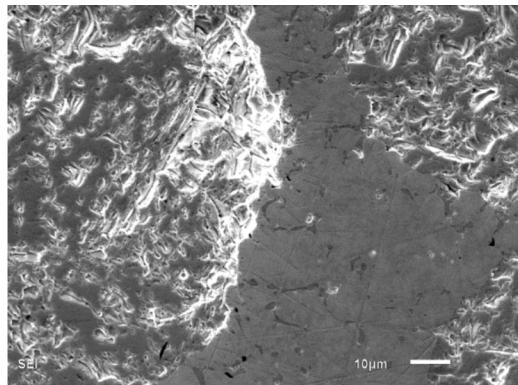


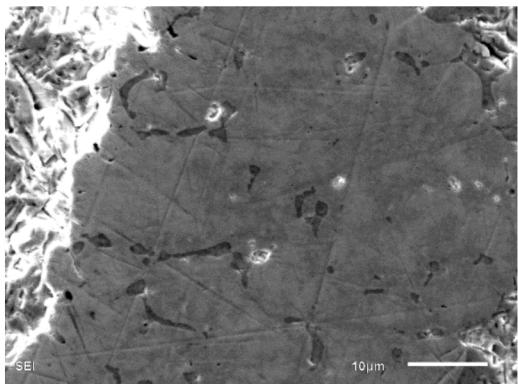
Figure 17. SEM images.



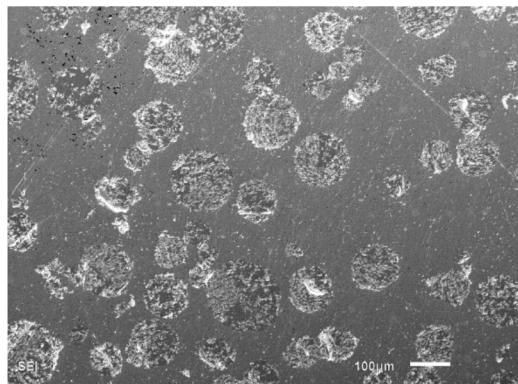
NASA-SPS-1750C-005 ( $\times$  500)



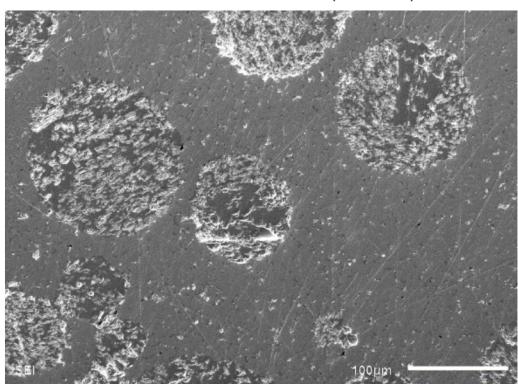
NASA-SPS-1750C-005 ( $\times$  1,000)



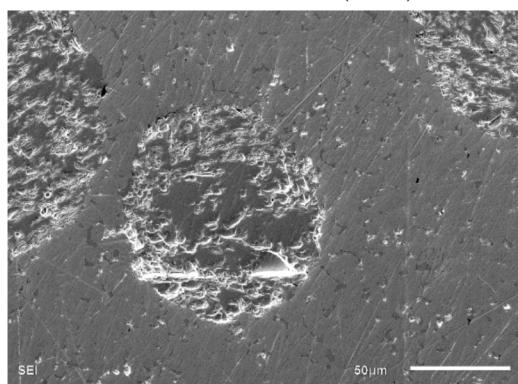
NASA-SPS-1750C-005 ( $\times$  2,000)



NASA-SPS-1700C-005 ( $\times$  100)



NASA-SPS-1700C-004 ( $\times$  250)



NASA-SPS-1700C-004 ( $\times$  500)

Figure 17. SEM images (Continued).

### B.3 Energy Dispersive X-Ray Spectroscopy Images

Figure 18 shows EDS images of NASA-SPS-1750C-004. Figure 19 shows EDS images of NASA-SPS-1700C-004.

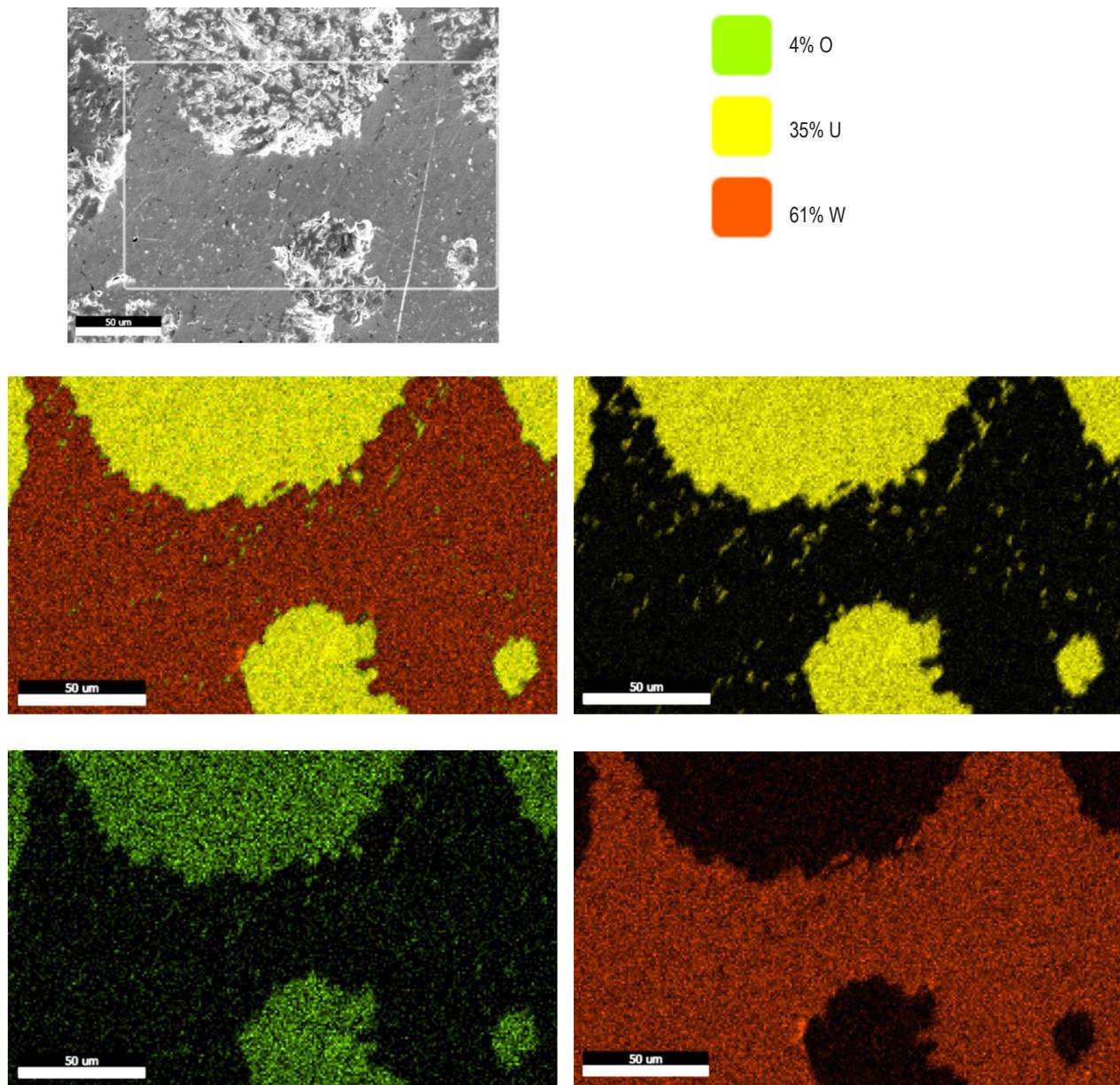


Figure 18. EDS images of NASA-SPS-1750C-004.

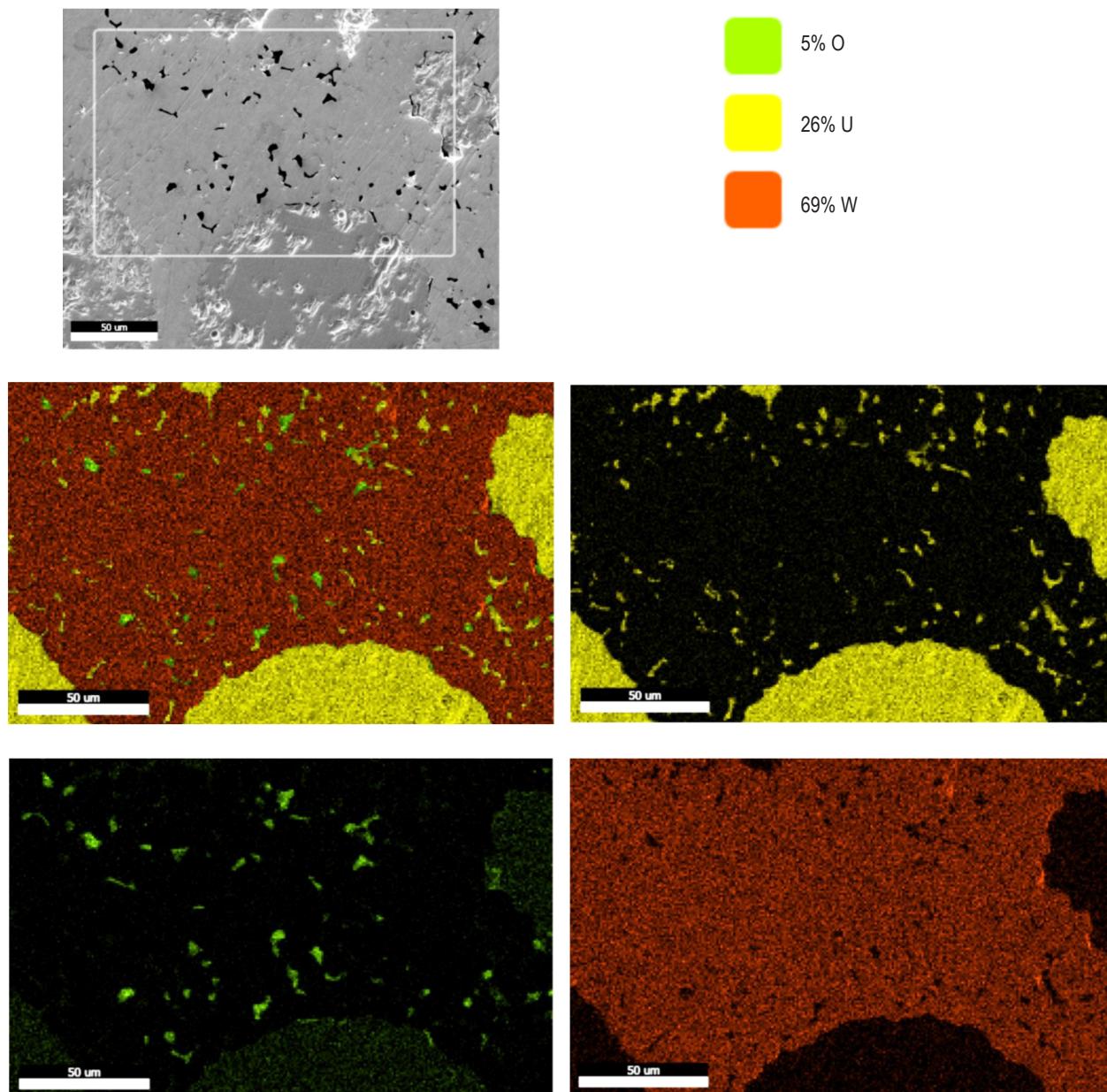


Figure 19. EDS images of NASA-SPS-1700C-004.

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